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The Behavior of Microdisk and Microring Electrodes.  
Mass Transport to the Disk in the Unsteady State:  
Coupled Chemical Reactions

by

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Prepared for publication in J. Electroanal. Chem.

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# ABSTRACT

In this work, we discuss the solution of the differential equations in the cylindrical coordinate system for the case of a coupled CE reaction mechanism at a finite disk electrode. We use the properties of discontinuous integrals of Bessel functions to treat the mixed boundary conditions at the electrode surface and the surrounding insulator. The results are compared to previous analyses and we also make comparisons of various estimates of the chronoamperometric response for constant surface concentration conditions.

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## The behavior of microdisk and microring electrodes. Mass transport to the disk in the unsteady state

### The effects of coupled chemical reactions: the CE mechanism

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#### ABSTRACT

In this work, we discuss the solution of the differential equations in the cylindrical coordinate system for the case of a coupled CE reaction mechanism at a finite disk electrode. We use the properties of discontinuous integrals of Bessel functions to treat the mixed boundary conditions at the electrode surface and the surrounding insulator. The results are compared to previous analyses and we also make comparisons of various estimates of the chronoamperometric response for constant surface concentration conditions.

#### INTRODUCTION

The application of microdisk electrodes to the investigation of the kinetics of chemical reactions in solution coupled to the electrode reactions (CE, ECE, and DISP reactions) has been investigated recently [1,2]. It was shown that the dimensions of the electrode become a parameter of the investigation, these dimensions in effect probing the reaction layer at the electrode surface. An interesting aspect of these measurements is that it becomes possible to differentiate between alternative reaction paths such as the ECE and DISP1 mechanism by combining measurements using microelectrodes with those using conventional planar electrodes; rate parameters can be derived from "working curves", e.g. of the effective number of electrons transferred as a function of the inverse of the radius of the electrode.

An approximate method of data analysis was used in these investigations. For example, for the CE mechanism



the flux of B to the surface was obtained from the solution of the differential equation in the spherical coordinate system in the steady state

$$D \frac{d^2 c}{dr^2} + \frac{2D}{r} \frac{dc}{dr} + k_1 - k_2 c = 0 \quad (1)$$

rather than that for the cylindrical coordinate system appropriate to the microdisk electrode

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial r^2} + \frac{D}{r} \frac{\partial c}{\partial r} + D \frac{\partial^2 c}{\partial z^2} + k_1 - k_2 c = 0 \quad (2)$$

in the steady state. The solutions obtained for a microsphere were then corrected to those for a microdisk by assuming that the same scale factor holds in these experiments as for the measurements of the voltammetric curve for a reversible electrode reaction, viz.

$$a_{\text{disk}} = \frac{4}{\pi} a_{\text{sphere}} \quad (3)$$

where  $a_{\text{disk}}$  and  $a_{\text{sphere}}$  are the relevant electrode radii.

We have shown recently that it is possible to develop systematic algebraic analyses of a variety of the usual types of electrochemical experiments (chronopotentiometry [3], chronoamperometry [4], ac impedance [5]) by appropriate solutions of the differential equations in the cylindrical coordinate system. In the derivation of these solutions we have made use of the properties of discontinuous integrals of Bessel functions to take account of the mixed boundary conditions at the electrode surface, viz. prescribed concentration or flux over the electrode surface, zero flux over the surrounding insulator surface. In this paper we explore the application of the same approach to the analysis of the kinetics of chemical reactions coupled to electrode reactions using the CE process (i) and (ii) to illustrate the method of calculation.

#### THEORETICAL CONSIDERATIONS

In the simplest method of investigating the kinetics of the coupled chemical reactions (i) and (ii), a sufficiently high overpotential is applied to the electrode surface to reduce the surface concentration of B to zero. The rate of the reaction then becomes controlled by the kinetics of reaction (i); species A is assumed to be present at sufficiently high concentrations that we need only consider the concentra-

tion distribution of species B, eqn. (2). It should be noted that it is relatively straightforward to achieve such conditions with microelectrodes (and of pseudo-first-order reaction conditions in second-order reactions) in view of the high rates of steady state mass transfer to the electrode surface [1,2].

We therefore require the solution of eqn. (2) subject to the condition

$$c = c^\infty = k_1/k_2 \quad r = \infty, z = \infty, \text{ all } t \quad (4)$$

and with

$$c = 0 \quad 0 < r < a, z = 0, t > 0 \quad (5)$$

$$\partial c / \partial z = 0 \quad r > a, z = 0, t > 0 \quad (6)$$

We have pointed out that the application of condition (5) to the solution of the mass transfer problem for the chronoamperometric response is difficult and these difficulties also apply to the present case. Instead we consider that the electrode is subjected to a constant uniform flux  $-Q$ , i.e.,

$$D \partial c / \partial z = -Q \quad 0 < r < a, z = 0, t > 0 \quad (7)$$

and we evaluate the average concentration of B over the electrode surface and then determine the strength of  $Q$  required to make this average concentration zero. It should be noted that the constant surface flux condition is in fact likely to be a very good approximation for the investigation of coupled chemical reactions since the rate will be controlled by step (i). Provided the concentration of A is uniform over the surface,  $Q$  will be uniform over the surface.

Laplace transformation of eqn. (2) subject to condition (4) gives

$$\frac{\partial^2 \bar{c}}{\partial r^2} + \frac{1}{r} \frac{\partial \bar{c}}{\partial r} + \frac{\partial^2 \bar{c}}{\partial z^2} - \left( q^2 + \frac{k_2}{D} \right) \bar{c} + \frac{k_1}{D} \left[ \frac{1}{k_2} + \frac{1}{s} \right] = 0 \quad (8)$$

where  $q = (s/D)^{1/2}$ .

The solution of eqn. (8) consists of the Particular Integral  $k_1/k_2 s$  and the Complementary Function which must be found from

$$\frac{\partial^2 \bar{c}_{CF}}{\partial r^2} + \frac{1}{r} \frac{\partial \bar{c}_{CF}}{\partial r} - \left( q^2 + \frac{k_2}{D} \right) \bar{c}_{CF} = 0 \quad (9)$$

By analogy to the discussion of the previous examples of electrochemical experiments at microdisk electrodes [3-5], we note that the substitution

$$\bar{c}_{CF} = \bar{v} \left[ -f \left( \lambda, q, \frac{k_2}{D} \right) z \right] \quad (10)$$

converts eqn. (9) into

$$\frac{\partial^2 \bar{v}}{\partial r^2} + \frac{1}{r} \frac{\partial \bar{v}}{\partial r} + \alpha^2 \bar{v} = 0 \quad (11)$$

with

$$\alpha^2 = \left[ f\left(\lambda, q, \frac{k_2}{D}\right) z \right]^2 - \left( q^2 + \frac{k_2}{D} \right) \quad (12)$$

The solution is then

$$\bar{c} = \frac{k_1}{k_2 s} - \int_0^\infty g\left(\lambda, q, \frac{k_2}{D}\right) \exp\left[ f\left(\lambda, q, \frac{k_2}{D}\right) z \right]^2 J_0(\alpha r) d\alpha \quad (13)$$

where the function  $g(\lambda, q, k_2/D)$  must be chosen to satisfy the Laplace transforms of eqns. (6) and (7).

$$\frac{d\bar{c}}{dz} = 0, \quad r > a, z = 0 \quad (14)$$

$$\frac{d\bar{c}}{dz} = -\frac{Q(s)}{D} \quad 0 < r < a, z = 0 \quad (15)$$

We again make use of the discontinuous integral

$$\int_0^\infty J_0(\alpha r) J_1(\alpha a) d\alpha = \begin{cases} 0 & r > a \\ 1/2a & r = a \\ 1/a & r < a \end{cases} \quad (16)$$

and with the particular form

$$\left[ f\left(\lambda, q, \frac{k_2}{D}\right) \right]^2 = \alpha^2 + q^2 + \frac{k_2}{D} \quad (17)$$

We obtain

$$\begin{aligned} \bar{c} &= \frac{k_1}{k_2 s} - \frac{Q(s)a}{D} \int_0^\infty \exp\left[ -\left( \alpha^2 + q^2 + \frac{k_2}{D} \right) z^{1/2} \right] J_0(\alpha r) J_1(\alpha a) \\ &\quad \times \frac{d\alpha}{\left( \alpha^2 + q^2 + \frac{k_2}{D} \right)^{1/2}} \end{aligned} \quad (18)$$

and, at  $z = 0$ ,

$$\bar{c} = \frac{k_1}{k_2 s} - \frac{Q(s)a}{D} \int_0^\infty J_0(\alpha r) J_1(\alpha a) \frac{d\alpha}{\left( \alpha^2 + q^2 + \frac{k_2}{D} \right)^{1/2}} \quad (19)$$

We derive the average surface concentration over the surface of the disk

$$\begin{aligned} \bar{c}_{Av} &= \frac{k_1}{k_2 s} - \frac{Q(s)a}{D} \int_0^a \int_0^\infty J_0(\alpha r) J_1(\alpha a) \frac{r dr d\alpha}{\left( \alpha^2 + q^2 + \frac{k_2}{D} \right)^{1/2}} \\ &= \frac{k_1}{k_2 s} - \frac{2Q(s)}{D} \int_0^\infty [J_1(\alpha a)]^2 \frac{d\alpha}{\alpha \left( \alpha^2 + q^2 + \frac{k_2}{D} \right)^{1/2}} \end{aligned} \quad (20)$$

and, on setting the average concentration at the surface equal to zero, we obtain

$$Q(s) = \frac{k_1 D / 2k_2 s}{\int_0^\infty [J_1(\alpha a)]^2 \frac{d\alpha}{\alpha(\alpha^2 + s^2 + k_2/D)^{1/2}}} \quad (21)$$

If we define

$$\beta = \frac{D^{1/2} \alpha}{(s + k_2)^{1/2}} \quad (22)$$

eqn. (21) can be written

$$Q(s) = \frac{\frac{k_1 a}{2k_2} \cdot a \left( \frac{s + k_2}{D} \right)^{1/2}}{\frac{a^2 s}{D} \int_0^\infty \left[ J_1 \left( \beta a \left( \frac{s + k_2}{D} \right)^{1/2} \right) \right]^2 \frac{d\beta}{\beta(\beta^2 + 1)^{1/2}}} \quad (23)$$

We therefore obtain a family of curves in the  $s$ -domain of  $Q(s)$  as a function of  $(a^2 s/D)$  each determined by the given value of the parameter  $(a^2 k_2/D)$ . Numerical inversion gives the responses in the  $t$ -domain. Here we restrict attention to the steady state limit

$$Q_{t \rightarrow \infty} = \frac{\frac{k_1 a}{2} \cdot \left( \frac{D}{a^2 k_2} \right)^{1/2}}{\int_0^\infty \left[ J_1 \left( \beta \left( \frac{a k_2^{1/2}}{D^{1/2}} \right) \right) \right]^2 \frac{d\beta}{\beta(\beta^2 + 1)^{1/2}}} \quad (24)$$

It is readily confirmed that this result also follows directly from eqn. (2) in the steady state provided we use the substitutions

$$c = v \exp \left[ - \left( \alpha^2 + \frac{k_2}{D} \right)^{1/2} z \right] \quad (25)$$

and

$$\beta = D^{1/2} \alpha / k_2^{1/2} \quad (26)$$

Equation (24) is identical to the chronoamperometric response [4] provided we replace  $(c^\infty - c_{Av})$  by  $k_1$  and  $s^{1/2}$ , by  $k_2^{1/2}$ , i.e. we obtain

$$Q^{1/2} = \frac{k_1 a}{2} \Phi_2 \left( \frac{a k_2}{D^{1/2}} \right) \quad (27)$$

The result of the approximate method of analysis (see above) can be written in the form

$$Q = \frac{k_1 a}{2} \cdot \frac{8}{\pi} \left( \frac{D}{a^2 k_2} \right) \left\{ 1 + \frac{\pi}{4} \left( \frac{a^2 k_2}{D} \right)^{1/2} \right\} \quad (28)$$



TABLE 1

Comparisons of the various results for the CE flux and diffusion limited current flux

$\frac{a^2 k_2}{D}$	Exact eqn. (27)	Equation (28)	Equation (29)	(eqn. 29) - (eqn. 27) (eqn. 27)	(SS) <sup>a</sup> - (eqn. 27) (eqn. 27)
0.01	23701	25664	23747	0.001943	0.000834
0.015	10564	11451	10595	0.002901	0.001248
0.02	5960.1	6466.2	5983.0	0.003849	0.001660
0.03	2664.1	2896.1	2679.1	0.005717	0.002476
0.04	1507.5	1641.5	1518.9	0.007547	0.003284
0.05	970.42	1058.6	979.49	0.009341	0.004082
0.06	677.82	740.69	685.34	0.011098	0.004872
0.07	500.87	548.26	507.29	0.012821	0.005653
0.08	385.69	422.89	391.29	0.014508	0.006425
0.09	306.50	336.60	311.45	0.016162	0.007189
0.1	249.69	274.65	254.12	0.017781	0.007945
0.2	66.028	73.662	68.158	0.032251	0.015056
0.3	30.987	34.961	32.348	0.043929	0.021421
0.4	18.374	20.915	19.353	0.053256	0.027117
0.5	12.376	14.186	13.126	0.060606	0.032211
0.6	9.0306	10.407	9.6292	0.066293	0.036765
0.7	6.9609	8.0540	7.4522	0.070585	0.040833
0.8	5.5832	6.4789	5.9947	0.073709	0.044462
0.9	4.6150	5.3660	4.9650	0.076854	0.047696
1.0	3.9053	4.5465	4.2067	0.077185	0.050573
2.0	1.4186	1.6366	1.5143	0.067456	0.065799
3.0	0.83920	0.94961	0.87862	0.046943	0.067957
4.0	0.59278	0.65915	0.60990	0.028887	0.065204
5.0	0.45767	0.50186	0.46436	0.014619	0.060779
6.0	0.37258	0.40407	0.37387	0.003464	0.056015
7.0	0.31414	0.33768	0.31245	-0.005360	0.051451
8.0	0.27152	0.28979	0.26813	-0.012472	0.047287
9.0	0.23908	0.25366	0.23470	-0.018303	0.043569
10.0	0.21356	0.22546	0.20862	-0.023153	0.040282
12.0	0.17599	0.18435	0.17058	-0.030741	0.034831

<sup>a</sup> Simulation result from Shoup and Szabo, reference (7).

The result, however, applies for constant surface concentration whereas eqn. (27) assumes a constant surface flux. The correction to the latter condition will be of the order of the ratio of the mass transfer coefficients for constant flux and constant concentration,  $3\pi^2/32$ , i.e. we predict the relation

$$Q = \frac{k_1 a}{2} \cdot \frac{3\pi}{4} \left( \frac{D}{a^2 k_2} \right) \left( 1 + \frac{\pi}{4} \left( \frac{a^2 k_2}{D} \right)^{1/2} \right) \quad (29)$$

Table 1 compares values of  $2Q/k_1 a$  for the various predictions (27)–(29) while Fig. 1 plots the percentage deviation between the predictions (29) and (27). It can be

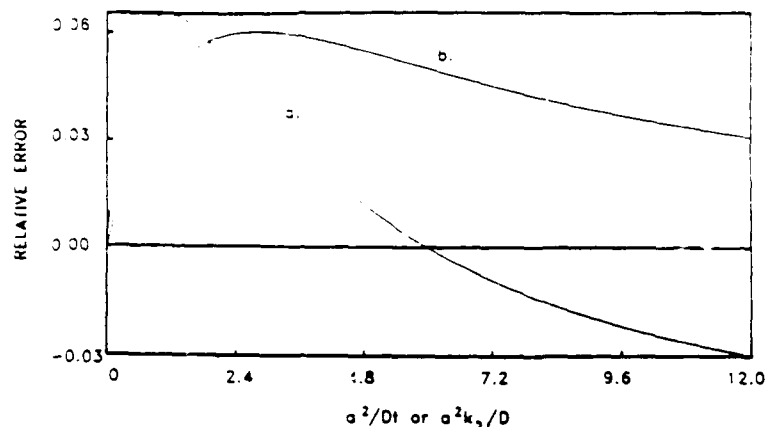


Fig. 1. Plot of the relative errors with respect to the exact CE result (27) or the exact chronoamperometric result (7) for (a) the CE approximation in eqn. (29) and (b) for the chronoamperometric approximation in eqn. (33).

seen that the approximate method gives results which are in fact very close to the result (27).

#### CONCLUSION

The present series of papers (see also refs. 3-5) has shown that it is possible to analyze a wide range of electrochemical experiments at microdisk electrodes by making use of the properties of discontinuous integrals of Bessel functions. In this analysis we have made extensive use of the average concentration over the surface of the microdisk rather than solving the problems for controlled uniform concentrations over the surface. The latter approach (although possible) leads to considerable mathematical complications and results which frequently have to be approximated. It is of interest therefore, that the approximate procedure used previously for the discussion of coupled reactions in solution (in which the exact result for the problem for a microspherical electrode is scaled to that for the disk using eqn. (3) [3]) gives results which are close to those using the procedure developed in this series of papers. As a further illustration of this procedure, we note that the diffusion limited current density to a sphere (a special case of constant surface concentration)

$$I = nFDc^\infty/a_{\text{sphere}} + nFD^{11/2}c^\infty/\pi^{1/2}t^{1/2} \quad (30)$$

can be rescaled to that at the disk using eqn. (3).

$$I = 4nFDc^\infty/\pi a_{\text{disk}} + nFD^{11/2}c^\infty/\pi^{1/2}t^{1/2} \quad (31)$$

We therefore obtain the actual current at a disk of radius  $a_{\text{disk}}$  as

$$\pi a_{\text{disk}}^2 I = 4nFDc^\infty a_{\text{disk}} + nF\pi^{1/2} D^{1/2} c^\infty a_{\text{disk}} / t^{1/2} \quad (32)$$

The current transient normalized by the steady state current is

$$\frac{\pi I a_{\text{disk}}}{4nFDc^\infty} = 1 + \frac{\pi^{1/2}}{4} \left( \frac{a_{\text{disk}}}{D^{1/2} t^{1/2}} \right) \quad (33)$$

We note that eqn. (33) is close to the values predicted algebraically [6] or by simulation [7] the maximum deviation being +6.8% at  $a_{\text{disk}}/D^{1/2}t^{1/2} \approx 3$  this deviation decreasing at both high and low values of  $a/D^{1/2}t^{1/2}$ , Table 1 and Fig. 1. We note that a wide range of problems has already been analyzed in the spherical coordinate system and that, when solutions are not available, these are easy to obtain in view of the uniform accessibility of the surface of spherical electrodes. The approximate procedure may therefore prove to be generally useful.

#### ACKNOWLEDGEMENT

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#### GLOSSARY OF ADDITIONAL SYMBOLS USED (see also ref. 4)

- $I$  Current density,  $A\text{ cm}^{-2}$   
 $k_1, k_2$  Homogeneous rate constants,  $s^{-1}$   
 $\beta$  Parameter  $D^{1/2}\alpha/(s + k_2)^{1/2}$

$$\Phi_2 = \frac{\left( \frac{D}{a^2 k_2} \right)^{1/2}}{\int_0^\infty \left[ J_1 \left( \beta \left( \frac{a k_2^{1/2}}{D^{1/2}} \right) \right) \right]^2 \frac{d\beta}{\beta(\beta^2 + 1)^{1/2}}}$$

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